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IS 7739-5 (1976): Code of Practice for Preparation of Metallographic Specimens, Part 5: Iron and steel and their examination [MTD 22: Metallography and Heat Treatment]



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“Knowledge is such a treasure which cannot be stolen”

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IS : 7739 (Part V) - 1976

(Reaffirmed 1996)

Indian Standard

CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART V IRON AND STEEL AND THEIR EXAMINATION

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

*Indian Standard*CODE OF PRACTICE FOR PREPARATION
OF METALLOGRAPHIC SPECIMENS

PART V IRON AND STEEL AND THEIR EXAMINATION

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*Indian Standard***CODE OF PRACTICE FOR PREPARATION
OF METALLOGRAPHIC SPECIMENS****PART V IRON AND STEEL AND THEIR EXAMINATION****0. FOREWORD**

0.1 This Indian Standard was adopted by the Indian Standards Institution on 24 December 1976, after the draft finalized by the Metallography and Heat-Treatment Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The primary object of metallographic examination is to reveal the constituents and the structure of metals and their alloys by means of the microscope. Because of the diversity in available equipment, the wide variety of problems encountered, and the personal element, this standard gives for the guidance of the metallographer only those practices which experience has shown are generally satisfactory. This part covers the polishing, etching and examination of iron and steel. The other parts of this code are as follows:

Part I General features

Part II Electrolytic polishing

Part III Aluminium and its alloys and their examination

Part IV Copper and its alloys and their examination

Part VI Lead and its alloys and their examination

Part VII Magnesium and its alloys and their examination

Part VIII Nickel and its alloys and their examination

Part IX Gold, silver, platinum, palladium and their alloys and their examination

Part X Tin and its alloys and their examination

Part XI Zinc and its alloys and their examination

0.3 In the preparation of this standard assistance has been derived from:

ASTM E3-62. Standard methods of preparation of metallographic specimens. American Society for Testing and Materials.

Metals Handbook 1948. American Society of Metals.

1. SCOPE

1.1 This standard (Part V) covers the polishing, etching and examination of iron and steel.

1.2 For convenience, this standard is divided in two sections, namely:

- a) *Macroscopic examination* — In which the study is made at low magnification (usually 10 X or less).
- b) *Microscopic examination* — In which the study is made at comparatively high magnification and resolution.

1.3 A metallographic examination shall frequently include both a macroscopic and microscopic examination. However, there are many occasions on which either the macroscopic or microscopic study alone is sufficient to supply the desired information.

SECTION I MACROSCOPIC EXAMINATION OF STEEL

2. GENERAL

2.1 Macro-examination is an inspection procedure for revealing certain aspects of the quality and structure of a metal by subjecting it to the corrosive action of an etchant and examining it visually or at low magnification. This is usually done on steels and may be classified into two groups of study: one, the study of the surface characteristics of the material, and the other the study of physical and chemical nonhomogeneity of the material, for example, porosity, cracks, seams, segregation, scabs, laps, piping, laminations, etc, or the absence of this condition, as revealed by sections of the material.

2.2 Other applications of macro-etching in the fabrications of metals, are the study of flaw lines of weld structure, definition of weld penetration, dilution of filler metal by base metals, entrapment of flux, porosity and cracks in weld and heat-affected zones, etc. It is also used in the heat-treatment shop to determine location of hard or soft spots, tong marks, quenching cracks, case depth in shallow hardening steels, case depth in carburization of dies, effectiveness of stop off coatings in carburization, etc. In the machine shop, it may be used for the determination of grinding cracks in tools and dies.

3. SELECTION OF SAMPLE

3.1 A representative sample shall be selected with due consideration for the purpose of the test. The sample should usually be of a full section of the material and should be thick enough for easy handling and subsequent preparation. Normally, transverse cross-sectional samples in the form of flats discs at right angle to the axis shall be taken, and the cross-sectional dimension should not exceed 300 mm × 300 mm.

3.1.1 If necessary, stipulation should be made in material specification regarding identity of the sample, for example, cast number, ingot number, relative position of the sample, etc, as agreed to between the supplier and the purchaser.

3.2 During sample selection, consideration should be given to the stage of mechanical working at which the sample is selected. When macro-etching is used as an inspection procedure, sampling ought to be done in an early stage of manufacturing so that if the material is faulty, no further work is done, which may prove unnecessary and wasteful. However, care should also be taken not to select a sample so early that further working may introduce serious defects. For steels sample shall be taken after ingot breakdown and after most chances of bursts or flakes occurring have passed. Billets or blooms going into small sizes are sampled after initial breakdown. Materials for forging billets or die blocks is sampled near finish size. Sampling may be done systematically or on a random basis.

3.3 Samples may be cut from the whole by means of a saw, abrasive cut-off wheels or any other convenient machining operation. Gas-cutting may be used to cut samples from large sections, but the heat-affected zone of gas-cut samples shall be removed by machining or abrasive wheel cutting prior to etching. Suitable coolant shall be used to prevent overheating of specimens during abrasive cutting.

4. PREPARATION OF SPECIMENS

4.0 The required surface finish for specimens for macro-etching vary from saw-cut or machined surface to polished surfaces.

The degree of permissible surface roughness depends on the severity of the etchant and the type of examination being made.

4.1 If the surface characteristics of a material are under study, it is rather unusual to have any extensive preparation of the surface prior to macroscopic study. The surface is usually cleaned and degreased with carbon tetrachloride so that the subsequent etchant shall attack the surface of the material uniformly. With a hot-dipped or electroplated coating present on the surface of the material under study it is necessary to remove the coating metal to examine the underlying base metal. These protective metals are usually stripped by chemical solution; extreme care should be exercised so that the base metal is unattacked by the stripping solution.

4.2 In the study of materials for physical and chemical nonhomogeneity, it is usually necessary to section the material through a plane which should reveal the area of suspected heterogeneity. The degree of surface preparation to be given to the sample is largely dependent upon the type of etch to be used in the examination and the details to be revealed in the sample under test. In general, the quality of the surface finish must be higher as the severity of the etchant in chemical attack decreases. Conversely, with an intensive etch, the quality of the surface preparation may be lowered. Rough grinding on an abrasive wheel or belt or with coarse emery paper is usually sufficient, and even machined surfaces may sometimes prove

satisfactory for deep etching specimens for macroscopic examination. When less severe etching, such as with ammonium persulphate, is to follow, or in sulphur printing, a somewhat smoother finish may be desirable.

5. ETCHING FOR MACROSCOPIC EXAMINATION

5.1 Table 1 gives the etching reagents which are commonly recommended for the macroscopic examination of iron and steel.

5.1.1 Table 2 gives the recommended methods of macroetching for specific purposes.

5.2 In deep etching, hot solutions of hydrochloric acid, sulphuric acid, or mixtures of these acids, are generally used. Deep etching in hot, strong solutions of these acids requires considerable care in the time of exposure of the material being etched, since the attack is frequently very rapid. It is very easy to over-etch materials and thus mask the interpretation that would be gained from an accurately etched specimen. Likewise, the intensity of a local defect should be given careful consideration if viewed after deep etching, since the chemical attack will exaggerate the original condition tremendously. A deep etch is frequently used to disclose physical and chemical nonhomogeneity, such as porosity, cracks, seams, segregation, etc. Ammonium persulphate, Stead's reagent, Fry's reagent, and Humfrey's reagent are less drastic in attack than the deep etching reagents and are used for grain structure development, detection of phosphorus banding, detection of strain lines, and dendritic segregation, respectively.

5.3 After etching, the sample shall be washed in warm water or running water, using a stiff brush to remove all traces of rust. They should be dried with alcohol and clean air, and examined immediately afterwards.

5.3.1 For photographic purposes, increased contrast is frequently obtained by washing after etching and applying glycerin to the etched surface.

5.3.2 A solution of one part of syrupy phosphoric acid to one part of water, to which a little sugar has been added, is sometimes applied to the dried face to delay rusting. Dipping in an alkaline solution before drying may be helpful. Dried surfaces may be protected with oil or grease or with a transparent lacquer.

6. SULPHUR PRINTING

6.1 This is a macroscopic method for revealing sulphur segregation and the size and distribution of the sulphides.

6.2 The surface, to be tested for sulphur distribution shall be reasonably smooth and free from foreign matter such as dirt and grease. Grind the surface on No. 00 or No. 000 emery paper and wash thoroughly in water to produce a surface satisfactory for taking a sulphur print.

Place a sheet of photographic paper (preferably matt finish, to prevent slipping), which has been soaked in a 2 percent aqueous solution of sulphuric acid for approximately 3 or 4 minutes, on the surface of the specimen and gently smooth out with a squeeze print roller so as to eliminate all entrapped air bubbles between the paper and the specimen surface. The paper shall be allowed to remain in contact with the specimen surface for 1 or 2 minutes. After that period, the paper shall be removed, rinsed, fixed in sodium thiosulphate (hypo), washed and dried in the usual manner.

Yellow or brown specks on the paper indicate the presence of sulphides in the specimen.

6.2.1 Any surface, if once used for taking on sulphur print, shall not be used again for taking another sulphur print. If more prints are desired from the same section at least 3 mm of steel should be removed prior to making another print.

6.2.2 Transparent silver bromide paper may be used to give a sulphur print transparency from which contact prints in any number may be made on ordinary photographic paper.

6.3 The specimen may also be tested by pressing for 2 to 4 minutes upon gelatin paper which has been treated with solution No. 1* and yellow cadmium acetate is precipitated. By immersion for 2 to 3 minutes in solution No. 2†, this is transformed to the dark brown to black copper sulphide.

7. PHOSPHORUS PRINTING

7.1 This is a macroscopic method for revealing the distribution of phosphorus on the surface of steel.

7.2 The surface of the specimen shall be prepared by grinding or on No. 000 emery paper and cleaned after grinding by washing in warm running water and carefully drying.

7.2.1 A photographic or filter paper is first soaked in a solution consisting of ammonium molybdate (5 g per 100 ml of water) to which 35 ml of nitric acid (r.d. 1.2) has been added. The paper is then removed from the reagent, drained nearly free of excess solution, and applied directly to the prepared surface of the metal, care being taken to effect intimate contact between the paper and the surface. The paper is allowed to remain in contact with the surface for approximately 5 minutes.

*Solution No. 1:

25 g of cadmium acetate plus 200 ml of acetic acid (80 percent) to which is added a solution of 500 ml of sulphuric acid (r.d. 1.84) in 950 ml of water.

†Solution No. 2:

120 g of copper sulphate plus 880 ml of water plus 120 ml of sulphuric acid (r.d. 1.84).

7.2.2 The print is then removed from the specimen and developed for approximately 4 minutes in a developer made up of 5 ml of saturated stannous chloride solution, 50 ml hydrochloric acid, 100 ml water and 1 g alum.

7.2.3 Development of a blue colour in the print indicates the location of phosphorus rich areas on the surface of the metal, and by noting the relative intensity of the blue colour, the amount of phosphorus present may be estimated. The intensity of colour increases with increased amounts of phosphorus.

8. LEAD PRINTING

8.1 This is a macroscopic method for revealing the lead distribution on the surface of a steel specimen.

8.2 The surface of the specimen shall be prepared by grinding on No. 000 emery paper and cleaned thoroughly in running water, degreased with a suitable solvent and dried.

8.2.1 Soak a caustic resistant filter paper in 5 percent caustic soda solution for 2 minutes. Place the soaked filter paper over the prepared surface and ensure through air-free contact between the paper and the specimen by means of a roller squeezer or by suitable application of pressure.

8.2.2 Remove the filter paper after maintaining the contact for about 4 minutes and then immerse it in 5 percent sodium sulphide solution for 1 minute. Take out the filter paper and wash in distilled water.

8.2.3 The brownish to dark spots indicate lead distribution in the examined surface.

8.3 It is difficult to preserve the lead prints, unless immediately photographed, preferably in colour.

SECTION 2 MICROSCOPIC EXAMINATION OF IRON AND STEEL

9. PREPARATION OF SPECIMENS

9.0 Recommended methods of selection, size, cutting, cleaning and mounting of specimens are given in IS : 7739 (Part I)-1975*. Recommended methods of electrolytic polishing are given in IS : 7739 (Part II)-1975†.

*Code of practice for preparation of metallographic specimens: Part I General features.

†Code of practice for preparation of metallographic specimens: Part II Electrolytic polishing.

9.1 A flat surface on the specimen may be obtained by filing, machining, or grinding on stones or motor-driven abrasive belts. In filing, the specimen should be pushed against the cutting edges of the file with the file in a fixed position. The motion of the specimen against the file should be in one direction only, since cutting action on both the forward and backward strokes shall tend to round the edges of the specimen. One of the most effective and rapid methods of facing a specimen is by the use of a motor-driven abrasive belt. Grinding wheels are effective but require considerable attention so that the grinding surface is true at all times. Extreme care should be exercised during grinding so that the specimen does not become heated and thus temper the steel which is to be examined. It has been demonstrated that grinding may raise the temperature of the surface layer of the specimen to 1 000°C.

9.2 The second stage of specimen preparation is the grinding with successively finer abrasive materials until the abrasive marks produced are fine enough to be removed readily by a final polishing operation.

9.3 The grinding operation wet or dry may be done either by hand or by power driven wheels if abrasive papers are used. Emery or carborundum papers are used in four or five degrees of fineness of grit. The commonly used papers are No. 2, 1, 0, 00, and 000. It is frequently not necessary to use the coarsest or No. 2 paper, but this is determined by the degree of surface obtained on the preliminary facing. If powder driven disks are used, the question of revolutions per minute is encountered. Actually the cutting speed is a function of linear speed; therefore it is impossible to name the revolutions per minute to be used on a disk since the linear speed varies from the centre of the disk to the periphery. The grinding speeds of the disks usually vary from 500 to 1 000 rev/min. The grinding on each paper should be done in one direction and at 90° to that of the preceding paper or stage. (For details of grit number of abrasive papers, see IS : 715-1976*. A comparative chart of grit numbers of abrasive grains is given at Appendix A.)

9.4 The grinding operation may be done on paraffined disks or on paraffin impregnated billiard cloth or canvas. The main advantage of using paraffined disks or impregnated cloths is the preservation of nonmetallic inclusions in the polished surface and the retention of graphite in cast iron.

9.5 The grinding operation may also be done by using lead or lead alloy laps. In a lead-lapped disk, the abrasive is firmly fixed in the soft metal, and it is claimed that there is less tendency to 'drag out' nonmetallic inclusions.

9.6 Rough polishing, which follows grinding, may be considered as a final grinding operation. The success of the polishing operation may largely be

*Specification for coated abrasives (*third revision*).

determined by this step. This may be done by any one of the following methods:

- a) Grinding on billiard cloth or canvas with aluminium oxide of grit No. 600,
- b) Grinding on fine abrasive papers such as No. 0000 or No. 600 grit silicon carbide, and
- c) Grinding with billiard cloth impregnated with paraffin.

9.7 The final polishing technique may be determined largely by the intent of the examination. If the type and amount of nonmetallic inclusions are to be studied, the final polish should be done on a disk with a hard, pileless cloth such as a good grade of heavy cotton or linen, the dull side of a heavy silk stain, or a good flat crepe. If the structure of the metal is to be studied at high power, a surface with fewer and less severe scratches, is required. Magnesium oxide or levigated suspension of γ -aluminium oxide are the common finishing powders. Finishing speeds are fairly slow in comparison with grinding.

While using magnesium oxide for final polishing, a small quantity of fresh, dry powder is applied to the lapping cloth and a thin paste is formed by adding sufficient quantity of distilled water. The paste is then worked into the cloth fibres with the finger tips, and after so charging the cloth and during subsequent polishing, additional distilled water is added when necessary to keep the lap moist.

The γ -aluminium oxide, used for final polishing, should have a uniform particle size of less than $0.1 \mu\text{m}$, and the pH of the suspending liquid shall be controlled between 8.3 and 10.0, by using phenolphthalein as indicator.

9.8 The polishing of cast iron specimens in which graphite is to be studied, and of steel specimens to be studied in the etched condition, should usually not be considered finished until the specimens have been etched and repolished a number of times to remove the surface layers that have been disturbed by the previous stages of polishing. Care may be required to avoid pitting of inclusions, if this process is repeated too often.

10. ETCHING FOR MICROSCOPIC EXAMINATION

10.1 In general, polished metal shows no trace of crystalline structure. Etching is used to clearly delineate the structure and to produce optical contrast among the various constituents. In Table 3 are given the etching reagents commonly recommended for microscopic examination of iron and steel.

10.2 The polished specimen should be thoroughly cleaned prior to etching. (The specimen should also be rubbed with caustic soda and washed in water.)

The cleanliness of the polished surface may be tested by immersing it in alcohol. If the surface is completely wetted by the alcohol it shall accept uniformly the attack of the etchant. If complete wetting of the surface is not obtained, the specimen should be cleaned in ether or some similar oil and grease solvent.

10.3 The cleaned specimen is immersed in the etchant. This immersion is frequently done by means of nickel or other corrosion-resisting tongs. The specimen is usually immersed with the polished face up so that the progress of the etching may be observed. During etching the specimen should be kept in motion to prevent gas bubbles from clinging to the surface of the steel and thus retarding a uniform attack by the etchant. The progress of the etching may generally be observed visually, but it should also be carefully timed. If the sample is not etched sufficiently on the first immersion the process may be repeated, although re-etching frequently causes staining. However, if the specimen is over-etched on the first immersion, or any following immersion the specimen should be repolished and sometimes even reground prior to etching again. Alternate polishing and etching are very often required, especially for high-alloy steels, in order to obtain a correct development of the microstructure.

10.3.1 Whenever the etching time is small, it is better to immerse the polished face only and take out the sample for examination of progress of etching. Only where the etching time is considerable, samples may be kept immersed in the etchant.

10.4 Following the etching, the specimen should be washed in water and immersed in alcohol and then dried in a blast of warm air. Cold water washing should be avoided since it retards the evaporation of the alcohol and usually tends to leave water spots on the surface of the etched specimen. During drying, the specimen should never be rubbed with cotton since it is usually fatal to the detail of the etch.

10.5 Polished and etched specimens, should be preserved if required in a desiccator or a sterilizer cabinet where dust, dirt, and moist gases from the atmosphere shall not collect on the surface.

10.5.1 Etched surfaces that are to be preserved for comparatively long periods of time should be coated thinly with a suitable lacquer. Lacquer coatings are best applied to prepared surfaces by partly immersing the specimen in a thinned solution of the lacquer, allowing the excess liquid to drain for a few seconds, and then permitting the lacquer to dry with the polished and etched surface in an up and horizontal position.

10.5.2 A thin coating of vaseline or neutral white mineral oil over the prepared surface is satisfactory in some cases for long time preservation, particularly when storage is made in a desiccator.

TABLE 1 ETCHING REAGENTS FOR MACROSCOPIC EXAMINATION OF IRON AND STEEL

(Clause 5.1)

No.	ETCHING REAGENT	COMPOSITION		REMARKS	USE
(1)	(2)	(3)		(4)	(5)
1.	Hydrochloric acid	HCl H ₂ O	50 ml 50 ml	Used at 71 to 82°C for 1 to 60 min depending on the size of sample, type of steel, and type of structure to be developed. Surface A or B	Shows segregation, porosity, cracks, depth of hardened zone in tool steel, etc. May produce cracks in strained steel
2.	Mixed acid	HCl H ₂ SO ₄ H ₂ O	38 ml 12 ml 50 ml	To be used as above for 15 to 45 min. Surface B or C	Same as for reagent No. 1
3.	Nitric acid	HNO ₃ H ₂ O	25 ml 75 ml	Used cold for large surfaces such as split ingots which may not conveniently be heated. Surface B or C	Same as for reagents No. 1 and 2
4.	Nitric acid	HNO ₃	0.5 to 1.0 percent in H ₂ O	Immerse 30 to 60 s after grinding specimen on 240-grit emery belt and thorough cleaning. Surface B or C	To show structure of welds
5.	Ammonium persulphate	(NH ₄) ₂ S ₂ O ₈ H ₂ O	10 g 90 ml	Surface should be rubbed with absorbent cotton during etching. Surface C	Brings out grain structure in cases of excessive grain growth, recrystallization at welds, flow lines, etc
6.	Ammonium persulphate with potassium iodide	A. (NH ₄) ₂ S ₂ O ₈ H ₂ O B. Same as A, plus KI C. Same as B, plus HgCl ₂ D. Same as C, plus H ₂ SO ₄	2.5 g 100 ml 1.5 g 1.5 g 15 ml	After grinding on No. 320 abrasive paper, swab for 15 min with solution A, then 10 min with B, then 5 min with C, and 5 min with D, finally washing with water and drying with alcohol	Shows dendritic macrostructure of cast iron

7. Stead's reagent	CuCl ₂ MgCl ₂ HCl Ethyl alcohol	2.5 g 10.0 g 5 ml Up to 250 ml	The salts are dissolved in the HCl with the addition of the least possible quantity of hot water. Surface <i>B</i> or <i>C</i>	Brings out phosphorus-rich areas and phosphorus bending. May be used also for general segregation
8. Fry's reagent	CuCl ₂ HCl H ₂ O	90 g 120 ml 100 ml	Most useful for mild steel, particularly bessemer and other high nitrogen steel. Before etching, sample should be heated to 200 to 250°C for 5 to 30 min, depending on the condition of the steel. During etching the surface should be rubbed with a cloth soaked in the etching solution. Wash in alcohol or rinse in HCl (1+1) after etching to prevent deposition of copper. Surface <i>B</i> or <i>C</i>	Shows up strain lines due to cold work
8A. Fry's reagent	CuCl ₂ HCl H ₂ O	45 g 180 ml 100 ml	Same as for reagent No. 6, may give more contrast. Specimen can be washed in water without depositing copper. Surface <i>B</i> or <i>C</i>	Same as for reagent No. 6
9. Nital	HNO ₃ Ethyl alcohol	5 ml 95 ml	Etch 5 min, followed by 1 sec in HCl (10 percent). Surface <i>B</i> or <i>C</i>	Shows cleanness, depth of hardening, carburized or decarburized surface, etc
10. Humfrey's reagent	Copper ammonium chloride HCl H ₂ O	120 g 50 ml 1 000 ml	Slight abrasion of surface after etching is recommended. Surface <i>B</i>	Develops dendritic segregation
11. Sulphuric acid	H ₂ SO ₄ H ₂ O	25 ml 75 ml	Use cold 8 to 16 h. Surface <i>B</i>	Shows dendritic pattern and flow lines

NOTE 1 — The use of concentrated reagents is intended unless otherwise specified.

NOTE 2 — Surface designations:

Surface *A* — Saw-cut or machined surface

Surface *B* — Average ground surface

Surface *C* — Polished surface

TABLE 2 MACROETCHING OF IRON AND STEEL FOR SPECIFIC PURPOSES

(Clause 5.1.1)

PURPOSE (1)	COMPOSITION (2)		REMARKS (3)	USE (4)
Blowholes	H ₂ SO ₄	10 ml	Use cold on large sections for 24 h. Surface B	Shows blowholes, porosity, pipe and inclusions
	H ₂ O	90 ml		
Carburised case	HNO ₃	3 ml	Etch at room temperature. Surface B	Shows depth of carburized case
	Ethyl alcohol	95 ml		
Contrast	Iodine	1 g	Use at room temperature. Surface B	Produces contrast for photographing
	KI	2 g		
	H ₂ O	10 ml		
Cracks	HNO ₃	5 ml	Etch at room temperature. Etches within 1/2 h. Surface B	Detects fatigue, service hardening and grinding cracks
	Ethyl alcohol	95 ml		
Cracks	Copper ammonium chloride	120 g	Etch first with a neutral solution to remove machine marks. Use cold for 20-30 min. Surface B	Produces a strong relief effect. Shows up dendrites and may indicate cracks
	HCl	50 ml		
	H ₂ O	1 000 ml		
Decarburization	HNO ₃	5 ml	Etch at room temperature. Surface C	The decarburized areas shall be light. Area not decarburized dark
	Ethyl alcohol	95 ml		
Defects	H ₂ SO ₄	10 ml	Use cold on large sections for 24 h. Surface B	Shows porosity, pipe, blowholes, and inclusions
	H ₂ O	90 ml		
Defects	H ₂ SO ₄	20 ml	Use near boiling for at least 1/2 h. Surface A	Shows general structure and defects
	HCl	10 ml		
	H ₂ O	30 ml		
Dendrites	H ₂ SO ₄	20 ml	Use near boiling for at least 1/2 h. Surface A	Shows general structure and defects
	HCl	10 ml		
	H ₂ O	30 ml		
Dendritic pattern	H ₂ SO ₄	25 ml	Use cold, 8 to 16 h. Surface B	Shows dendritic pattern and flow lines
	H ₂ O	75 ml		
Dendritic structure	FeCl ₃	40 g	Etch first with 10 percent nitric acid. Surface B	Shows dendritic structure
	CuCl ₂	3 g		
	HCl	40 ml		
	H ₂ O	500 ml		

Fibre	(NH ₄) ₂ S ₂ O ₈ H ₂ O	10-12 g 90 ml	Swab on freshly made solution for 1/2 min. Surface <i>B</i>	Shows fibre and grain contrast
Flow lines	H ₂ SO ₄ H ₂ O	25 ml 75 ml	Use cold, 8 to 16 h. Surface <i>B</i>	Shows flow lines and dendritic pattern
Flow lines	HCl H ₂ O	50 ml 50 ml	Use hot. Surface <i>A</i>	Shows flow lines
Grain contrast	(NH ₄) ₂ S ₂ O ₈ H ₂ O	10-20 g 90 ml	Swab on freshly made solution for 1 to 2 min. Surface <i>B</i>	Shows grain contrast and fibre
Grain contrast	CuCl ₂ HCl H ₂ O Ethyl alcohol	1.5 g 30 ml 95 ml 30 ml	Polish specimen carefully. Surface <i>C</i>	Shows good grain contrast
Grain size	(NH ₄) ₂ S ₂ O ₈ H ₂ O	10-20 g 90 ml	Swab on freshly made solution for 1 to 2 min. Surface <i>B</i>	Shows grain size especially in low-carbon steels, wrought iron, and welded sections
Hardness penetration	HNO ₃ Ethyl alcohol	5 ml 95 ml	Etch at room temperature on smooth ground surface. Surface <i>B</i>	Shows depth of hardness penetration of heat treated samples
Hardness penetration	HCl H ₂ O	50 ml 50 ml	Use hot 2 to 5 min. To preserve surface after etching, scrub in running water, dip in weak NH ₄ OH, dip in solution of soluble cutting oil, dry with a rag and compressed air. Surface <i>B</i>	Shows depth of hardening, especially carbon steels. Produces distinct contrast between martensitic and troostitic zones
Heterogeneity	HCl H ₂ O	50 ml 50 ml	Immerse specimen in hot solution form 1/2 to 1 h. Surface <i>A</i>	Shows heterogeneity in general
Heterogeneity	HCl	—	Use hot 100°C. Surface <i>A</i>	Shows heterogeneity in 3 to 3.5 percent nickel steels

NOTE — Surface designations:

Surface *A* — Saw-cut or machined surface

Surface *B* — Average ground surface

Surface *C* — Polished surface

(Continued)

TABLE 2 MACROETCHING OF IRON AND STEEL FOR SPECIFIC PURPOSES — *Contd*

PURPOSE (1)	COMPOSITION (2)		REMARKS (3)	USE (4)
Impurities in ferrite	CuCl ₂	1 g	Etch only well polished surface which has been thoroughly dried. Surface C	Plates out copper on ferrite containing impurities. Gives a good even etch
	SnCl ₂	0.5 g		
	FeCl ₃	30 g		
	HCl	30 ml		
	H ₂ O	500 ml		
	Ethyl alcohol	500 ml		
Inclusions	H ₂ SO ₄	10 ml	Use cold on large sections for 24 h. Surface B	Shows inclusions, porosity, pipe and blowholes
	H ₂ O	90 ml		
Inclusions	H ₂ SO ₄	10-20 ml	Use hot. Surface A	Etches sulphide inclusions
	H ₂ O	80-90 ml		
Pipe and porosity	H ₂ SO ₄	10 ml	Use cold on large sections for 24 h. Surface B	Shows pipe, porosity, blowholes, and inclusions
	H ₂ O	90 ml		
Rail sections	HCl	45 ml	Etch near boiling point for 2 h. Surface A	Use for etching rail sections
	H ₂ SO ₄	15 ml		
	H ₂ O	5 ml		
Segregation	Picric acid	3 g	Etch at room temperature for 4 to 5 h. Surface B	Shows segregation
	Ethyl alcohol	100 ml		
Segregation	Picric acid	1 g	Use hot. Surface B	Shows segregation
	HCl	1 drop		
	Ethyl alcohol	25 ml		
Segregation	HNO ₃	10-15 ml	Etch at room temperature. Surface B	Shows heavy segregation
	H ₂ O or ethyl alcohol	85-90 ml		
Segregation	HNO ₃	5 ml	Etches within 1/2 h. Surface is black when etched. Surface B	Shows segregation in low carbon, low chromium nickel steels
	H ₂ O or ethyl alcohol	95 ml		
Segregation	HNO ₃	2-10 ml	Etch first with weak acid to remove machine marks and then increase concentration for structure. Surface B	Shows segregation in ingot sections and large pieces
	H ₂ O	90-98 ml		

Segregation	CuCl ₂	1 g	May be used for electrolytic etching. Surface <i>B</i>	Shows segregation
	Picric acid	0.5 g		
	HCl	1.5-2.5 ml		
	H ₂ O	10 ml		
	Ethyl alcohol	100 ml		
Segregation, carbide	HNO ₃	5 ml	Use on a polished, longitudinal section. For high-speed steel, etch until matrix is darkened. Surface <i>C</i>	The detect carbide segregations, particularly in high-speed steels
	Ethyl alcohol	95 ml		
Segregation, carbon and phosphorus	Copper ammonium chloride	5 g	Immerse finely ground, clean sample in solution for 1 min: wash with water and rub off copper. Surface <i>C</i>	Shows phosphorus and carbon segregation
	H ₂ O	60 ml		
Segregation, phosphorus	Picric acid	0.5 g	Etch at room temperature until staining occurs. Surface <i>C</i>	Uneven staining represents phosphorus segregation
	H ₂ O	100 ml		
Segregation, phosphorus	FeCl ₃	30 g	Polish as for microscopic work, use etch cold for 10 sec to 2 min. Surface <i>C</i>	Shows phosphorus segregation
	CuCl ₂	1 g		
	SnCl ₂	0.5 g		
	HCl	100 ml		
	H ₂ O	1 000 ml		
Segregation, phosphorus	CuCl ₂	10 g	Dissolve salts in small amount of hot water then add alcohol. Apply solution to polished surface drop by drop. Surface <i>C</i>	Area not coated by copper shows phosphorus segregation
	MgCl ₂	40 g		
	HCl	20 ml		
	Ethyl alcohol	1 000 ml		
Segregation, phosphorus	CuCl ₂	5 g	Etch for 1 min. Surface <i>C</i>	Shows phosphorus segregation
	MgCl ₂	4 g		
	HCl	1 ml		
	H ₂ O	20 ml		
	Ethyl alcohol	100 ml		

NOTE — Surface designations :

Surface *A* — Saw-cut or machined surface

Surface *B* — Average ground surface

Surface *C* — Polished surface

(Continued)

TABLE 2 MACROETCHING OF IRON AND STEEL FOR SPECIFIC PURPOSES — *Contd*

PURPOSE (1)	COMPOSITION (2)		REMARKS (3)	USE (4)
Segregation, phosphorus	CuCl ₂	4 g	Polish specimen carefully. Sur- face C	Shows phosphorus segregation
	HCl	20 ml		
	H ₂ O	40 ml		
	Ethyl alcohol	20 ml		
Segregation phosphorus	CuCl ₂	1.5 g	Immerse specimen for 90 sec or more. Surface C	Shows phosphorus segregation
	Ni(NO ₃) ₂	5 g		
	FeCl ₃	6 g		
	H ₂ O	12 ml		
Segregation, sulphide inclusions	H ₂ SO ₄	10-20 ml	Use hot. Surface A	Etches sulphide inclusions
	H ₂ O	80-90 ml		
Soft spots	HNO ₃	5 ml	Etch at room temperature. Surface B	Non-destructive test for hardened tools
	Ethyl alcohol	95 ml		
Soft spots	HCl	50 ml	Use cold on ground surface. Sur- face B	Shows soft spots
	H ₂ O	50 ml		
Strains	CuCl ₂	90 ml	Copper does not precipitate with this etch. Heat specimen to 205 to 245°C for 1/2 h, then etch ground surface for 1-3 min. Rub with powdered cupric chloride, then rinse with alcohol. Surface B	Shows strain lines
	HCl	120 ml		
	H ₂ O	100 ml		
Strains	CuCl ₂	6 g	Heat specimen to 200°C and immerse ground surface. Surface B	Shows strain lines
	FeCl ₃	6 g		
	HCl	10 ml		
	Ethyl alcohol	100 ml		

Strauss test	CuSO ₄	3 g	Specimen 12 × 25 × 100 mm is sensitized by actual welding, or artificially by heating to 480 to 700°C (generally 680°C). Immersed in the boiling cupric sulphate solution for 72 h. Specimen is then bent 180° around 12 mm pin. Cracking indicates disintegration has taken place due to migration of carbides to grain boundaries. Surface <i>B</i>	To measure intergranular corrosion of 18-8 corrosion resisting steel
	H ₂ SO ₄	10 ml		
	H ₂ O	87 ml		
Structure	HNO ₃	25 ml	Etch at room temperature. Surface <i>B</i>	Shows general structure
	H ₂ O	75 ml		
Structure	H ₂ SO ₄	20 ml	Use near boiling point for at least 1/2 h. Surface <i>A</i>	Shows general structure and defects
	HCl	10 ml		
	H ₂ O	30 ml		
Weld examination	HCl	50 ml	When testing large sections, the acid may be added by building a dam with paraffin wax around the part to be tested. Surface <i>A</i>	For testing the soundness of welds
	H ₂ O	50 ml		

NOTE — Surface designation:

Surface *A* — Saw-cut or machined surface

Surface *B* — Average ground surface

Surface *C* — Polished surface

TABLE 3 ETCHING REAGENTS FOR MICROSCOPIC EXAMINATION OF STEELS AND IRONS
(Only reagents of analytical grade should be used)

[(Clause 10.1)

No.	ETCHING REAGENT	COMPOSITION	REMARKS	USE
(1)	(2)	(3)	(4)	(5)
Group I General Reagents for Irons and Steels (Carbon, Low and Medium-Alloy Steel)				
1. Nitric acid (Nital)	HNO ₃ , colourless Ethyl or methyl alcohol, 95 percent or absolute (also amyl alcohol)	1-5 ml 100 ml	Etching rate is increased, selectivity decreased, with increasing percentage of HNO ₃ . Reagent No. 2 (picric acid) usually superior, 4 percent in amyl alcohol useful for grain boundary and contrast of low-carbon materials. Etching time, a few seconds to a minute	In carbon steel, (a) to darken perlite colonies, (b) to reveal ferrite boundaries, (c) to differentiate ferrite from martensite. The 1 percent solution is also suitable for uses (c), (d), (e) and (f) noted below for reagent No. 2
2. Picric acid (Picral)	Picric acid Ethyl or methyl alcohol, 95 percent or absolute (Use absolute alcohol only when acid contains 10 percent or more of moisture)	4 g 100 ml	More dilute solutions occasionally useful. Does not reveal ferrite grain boundaries as readily as No. 1. Etching time a few seconds to a minute or more	For all grades of carbon steels: (a) annealed, (b) normalized, (c) quenched, (d) quenched and tempered, (e) spheroidized, and (f) austempered. For all low-alloy steels attacked by this reagent
3. Hydrochloric and picric acid	HCl Picric acid Ethyl or methyl alcohol, 45 percent or absolute	5 ml 1 g 100 ml	Best results are obtained when the martensite is tempered for 15 min at 205 to 245°C	For revealing the austenite grain size in quenched, and quenched-and-tempered steels
4. Chromic acid	CrO ₃ H ₂ O	10 g 100 ml	Used electrolytically, the specimen as anode, stainless steel or platinum as cathode, 20 to 25 mm apart; 6 V usually used. Time of etching 30 to 90 s depending on specimen	For various structures except grain boundaries of ferrite. Attacks cementite very rapidly, austenite less rapidly, ferrite and iron phosphide very slowly if at all

- | | | | |
|-----------------|-----------|--|--|
| 5. Heat tinting | Heat only | Clean, dry, polished specimen heated face up on hot plate to 205 to 370°C. Time and temperature both have decided effects. Bath of sand or molten metal may be used | Pearlite first to pass through a given colour, followed by ferrite; cementite less affected, iron phosphide still less. Especially useful for cast irons |
| 6. Heat etching | Heat only | Specimen is heated 10 to 60 min at 815 to 1205°C in carefully purified hydrogen and should have no contact with scale or reducible oxides. After etching, specimen is cooled in mercury to avoid oxidation | For revealing austenitic grain size of polished specimens |

Group II General Reagents for Alloy Steels (Stainless and High-Speed Steels)

- | | | | | |
|--|---|---------------------------|--|---|
| 7. Ferric chloride and hydrochloric acid | FeCl ₃
HCl
H ₂ O | 5 g
50 ml
100 ml | Etch at room temperature | Structure of austenitic nickel and stainless steels |
| 8. Nitric acid (Nital) | HNO ₃ (colourless)
Ethyl or methyl alcohol
(95 percent or better) | 5 to 10 ml
90 to 95 ml | Etch at room temperature | General structure of high-speed tool steel |
| 9. Chrome regia | HCl
CrO ₃ solution (10 percent) in H ₂ O | 25 ml
5 to 50 ml | Activity is controlled by amount of chromic acid | Heat-treated 18 percent chromium, 8 percent, nickel stainless steels. Useful electrolytically, if diluted with 2 parts alcohol and 2 parts glycerin and applied for 20 to 60 s with 6 V |
| 10. Ferric chloride and nitric acid | Saturated solution of FeCl ₃ in HCl, to which a little HNO ₃ is added | | Use full strength | Structure of stainless steel |

NOTE — The use of concentrated reagents is intended unless otherwise specified.

(Continued)

NS — Contd

TECHNIQUES AND APPLICATIONS OF STEEPIC EXAMINER MICROSCOPIC REAGENTS FOR ETCHING

TABLE 3

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USERS		REMAION	COMPOSITING AGENT		No. ETCH REAC
(5)		(4)	(3)	(2)	(1) (2)
chromium base steels, and manganese steel. chromium alloys	Structure of iron water before alloys, high-speed results use austenitic manate polishing Etches nickel-given sufficient satisfactorily totally austeni- better results using reagent	farm specimen is 10 ml etching. For b) to 30 ml method of alter and etching. If 10 to 30 ml time, shall etch tic alloys, but are obtained by No. 10-C	HNO_3 HCl Glycerin	acids in 2rol 2	A. H E C 11. Mixed glyce
gh silicon alloys pe	Structure of hijn may be va- of Duriron tyal	mount of glycer 10 ml ried to suit met 20 ml 20 to 40 ml	HNO_3 HF Glycerin		B. F E C
chromium-nickel, um - manganese, r austenitic iron- se alloys	To etch iron may be varied if iron - chromio rapidly or and all other results employ chromium barnate polishing	mount of HCl n 10 ml reagent acts to 20 ml slowly. For best 20 ml method of alte 10 ml and etching	HNO_3 HCl Glycerin H_2O_2		C. F E C I
tainless steels and alloys. Carbide structure revealed etching time	For austenitic sally, the speci- high-nickel stainless steel or and general shode, about 25 depending on/ usually used. rbides in stain- be revealed in general struc- min. For study to 3 V may be easing the etch- nd improving	Jsed electrolytic 10 g men as anode, 100 ml platinum as cat mm apart; 6 V Precipitated ca less steels, may 10 to 15 s, the ture in about 1 of carbides, 1.5 used, thus incr ing time a control of etch	Oxalic acid H_2O	acid	12. Oxalic

13. Ammonium persulphate	$(\text{NH}_4)_2\text{S}_2\text{O}_8$ H_2O	10 g 90 ml	Used electrolytically like the oxalic acid solution, but acts more slowly, requiring longer than 15 s with 6 V	For fast-etching stainless steels
14. Cupric chloride and hydrochloric acid	CuCl_2 HCl Ethyl alcohol H_2O	5 g 100 ml 100 ml 100 ml	Used cold (Kalling's reagent)	For austenitic and ferritic steels, the ferrite being most easily attacked, and austenite attacked appreciably only when it has begun to decompose to martensite
15. Mixed acids and cupric chloride	HCl HNO_3 Saturate with cupric chloride and let stand for 20 to 30 min before use	30 ml 10 ml	Apply by swabbing	For stainless alloys and others high in nickel or cobalt
16. Nitric and acetic acids	HNO_3 Acetic acid	30 ml 20 ml	Apply by swabbing	For stainless alloys and others high in nickel or cobalt
17. Nitric and hydrofluoric acids	HNO_3 HF (48 percent) H_2O	5 ml 1 ml 44 ml	Used cold for about 5 min	For revealing general structure of austenitic stainless steel with avoidance of stain markings
18. Hydrochloric and nitric acids in alcohol	HCl HNO_3 Methyl alcohol	10 ml 3 ml 100 ml	Etch 2 to 10 min	To reveal the grain size of quenched, or quenched and tempered high speed steel
19. Hydrochloric acid in alcohol	HCl Alcohol	10 ml 90 ml	Used electrolytically for 10 to 30 s with 6 V; must be free from water to prevent tarnishing	For straight chromium and chromium-nickel steels
20. Lactic and hydrochloric acids in alcohol	Lactic acid HCl Alcohol	45 ml 10 ml 45 ml	Used electrolytically for 10 to 30 s with 6 V; does not tarnish	For chromium steels (4 to 30 percent chromium) or delta ferrite in austenitic stainless steels

NOTE — The use of concentrated reagents is intended unless otherwise specified.

(Continued)

TABLE 3 ETCHING REAGENTS FOR MICROSCOPIC EXAMINATION OF STEELS AND IRONS — Contd
(Only reagents of analytical grade should be used)

No.	ETCHING REAGENT	COMPOSITION	REMARKS	USE
(1)	(2)	(3)	(4)	(5)
Group II General Reagents for Alloy Steels (Stainless and High-Speed Steels)				
21.	Ferricyanide solution	Potassium ferricyanide KOH H ₂ O 30 g 30 g 60 ml	Must be fresh. Use boiling	To distinguish between ferrite and sigma phase in iron-chromium, iron-chromium-nickel, iron-chromium-manganese and related alloys. Colours: sigma phase, light blue; ferrite, yellow
22.	Cupric sulphate	CuSO ₄ HCl H ₂ O 4 g 20 ml 20 ml	Marble's reagent	Structure of stainless steels
23.	Hydrochloric and picric acids	HCl Picric acid Ethyl or methyl alcohol, 95 per cent or absolute 5 ml 1 g 100 ml	Same as No. 3	To etch many steels of the iron-chromium, iron-chromium-nickel and iron-chromium-manganese types
24.	Heat tinting	Heat only in air for 10 to 60 s at about 595 to 650°C	Carbides remain white and austenite darkens less rapidly than ferrite, on sections, preferably etched first with a chemical reagent	For austenitic stainless steel containing ferrite and carbide
Group III Segregation, Primary Structure and Strain Lines				
25.	Cupric chloride	A. CuCl ₂ MgCl ₂ HCl H ₂ O Alcohol, absolute 1 g 4 g 1 ml 20 ml 100 ml	Dissolve salts in least possible quantity of hot water. Etch for about 1 min repeating if necessary. Stead's reagent	For showing segregation of phosphorus or other elements in solid solution; copper tends to deposit first on areas lowest in phosphorus. The structure may be more clearly delineated by light hand-polishing to remove the copper deposit after etching

	B. CuCl_2	5 g	May be used cold. Etching time, about 10 s. Fry's reagent	To reveal strain lines and their microstructure, and precipitation hardening in steel
	HCl	40 ml		
	H_2O	30 ml		
	Ethyl alcohol	25 ml		
26. Modified ferric chloride	FeCl_3	30 g	Oberhoffer's reagent	For showing phosphorus segregation and dendritic structure
	CeCl_2	1 g		
	SnCl_2	0.5 g		
	HCl	50 ml		
	Ethyl alcohol	500 ml		
	H_2O	500 ml		
27. Alkaline chromate	CrO_3	16 g	Add NaOH slowly, and use when not over one day old, boiling at 118 to 120°C for 7 to 20 min	Shows oxygen segregation by darkening martensite rapidly, ferrite more slowly, and zones of high oxygen content much more slowly
	H_2O (distilled)	145 ml		
	NaOH	80 g		
Group IV Structure and Depth of Case of Nitrided Steels				
28. Cupric sulphate and cupric chloride	CuSO_4	1.25 g	Proportions should be accurate. Etch by immersion to avoid confusing edge effects	For showing total depth, structure, and various zones of nitrided chromium-vanadium steels and nitralloy
	CuCl_2	2.50 g		
	MgCl_2	10 g		
	HCl	2 ml		
	H_2O	100 ml		
	Dilute above solution to 1 000 ml with 95 percent ethyl alcohol			
29. Picric and nitric acids	Picric acid (4 percent) (No. 2)	10 parts	Best results are obtained when the specimen is annealed in lead at 800°C before etching	For depth of case and structure of nitralloy
	HNO_3 (4 percent) (No. 1)	1 part		
30. Nitric acid (Nital)	HNO_3	2 ml	Etch at room temperature	For structure and depth of case of nitrided steels
	Ethyl or methyl alcohol, 95 percent or absolute	100 ml		
31. Cupric sulphate	CuSO_4	4 g	Marble's reagent	Total depth of nitrided case
	HCl	20 ml		
	H_2O	20 ml		

NOTE — The use of concentrated reagents is intended unless otherwise specified.

(Continued)

TABLE 3 ETCHING REAGENTS FOR MICROSCOPIC EXAMINATION OF STEELS AND IRONS — *Contd*

(Only reagents of analytical grade should be used)

No.	ETCHING REAGENT	COMPOSITION	REMARKS	USE
(1)	(2)	(3)	(4)	(5)
Group V Reagents for Carbides, Phosphides, Nitrides and Tungstides				
32.	Sodium picrate (neutral)	Sodium picrate 1 g H ₂ O 100 ml (Wash salt well with alcohol to remove excess acid or alkali)	Use boiling. Etching time 20 min	Shows difference between phosphides and cementite; iron phosphide attacked, cementite unattacked
33.	Chromic acid and heat tinting	CrO ₃ 8 g H ₂ O 100 ml Followed by heat tinting	Etching in picric acid (No. 2) then for 1 min in chromic acid; heat tint by heating face up on hot plate at about 260°C for 1 min	Distinguishes between iron phosphide and cementite in phosphide eutectic of cast iron; iron phosphide is coloured darker
34.	Sodium picrate, alkaline	Picric acid 2 g NaOH 25 g H ₂ O 100 ml	Use boiling, 5 to 10 min, or preferably, electrolytically at room temperature; for the latter, specimen is anode, platinum or stainless steel is cathode; with 6 V, about 40 s is usually sufficient	Colours cementite, but not carbides high in chromium. In tungsten steels, iron tungstide (FeW) and iron tungsten carbide (Fe ₄ W ₃ C) are coloured more rapidly than cementite, but tungsten carbide is unaffected. Attacks sulfides. Delineates grain boundaries in hypereutectoid steels slowly cooled condition
35.	Hydrogen peroxide and sodium hydroxide	H ₂ O ₂ 10 ml NaOH 10 percent 20 ml solution in water	Should be fresh. Etching time 10 to 12 min	Attacks and darkens iron-tungstide in carbon-free tungsten alloys. When carbon is present this solution darkens the compound (FeW WC) in proportion to the amount of carbide present; tungsten carbide is darkened

36. Ferricyanide solution	A. $K_3Fe(CN)_6$ KOH H_2O	1 to 4 g 10 g 100 ml	Should be freshly made; etch 15 min in boiling solution. Seven grams of NaOH may be substituted for 10 g of KOH in either A or B	Differentiates between carbides and nitrides. Cementite is blackened, pearlite turned brown, and massive nitrides remain unchanged
	B. $K_3Fe(CN)_6$ KOH H_2O	10 g 10 g 100 ml	May be used cold, but preferably hot, should be freshly made, etching time 5 to 10 min. Murakami's reagent	Darkens carbide containing chromium, carbides, and tungstides in tungsten and high-speed steels. At room temperature colours ternary carbides (Fe_3W_3C or Fe_3W_2C) in a few seconds, iron tungstide (Fe_3W_4) in several minutes, and barely colours cementite
37. Sodium cyanide	NaCN H_2O	10 g 90 ml	Used electrolytically, the specimen as anode, cathode, similar material; about 25 mm apart 6 V (not less than 5). Etching time 5 min or more	Darkens carbides without attacking austenite or grain boundaries
38. Chromic acid	CrO_3 H_2O	10 g 100 ml	See No. 4 in Group 1.	Attacks carbides in stainless steels very rapidly, austenite less rapidly, and ferrite very slowly if at all. For various structures of stainless steels
39. Oxalic acid	Oxalic acid H_2O	10 g 100 ml	See No. 12 in Group II. If strongly etched general structure is revealed; therefore for study of carbides reduced voltage is used for etching, giving better control of etch	Reveals carbides in stainless steels

NOTE — The use of concentrated reagents is intended, unless otherwise specified.

(Continued)

TABLE 3 ETCHING REAGENTS FOR MICROSCOPIC EXAMINATION OF STEELS AND IRONS — Contd
(Only reagents of analytical grade should be used)

No.	ETCHING REAGENT	COMPOSITION		REMARKS	USE
(1)	(2)	(3)		(4)	(5)
Group VI Reagents for Detection of Temper Embrittlement					
40. Picric acid	Picric acid H ₂ O	2 g 100 ml	Etching time about 10 minutes. After etching, carefully remove the deposit under water by means of a cotton wool pad	A specimen of the steel, which appears to be temper brittled is etched with any one of the reagent No. 35, 36 or 37, simultaneously with a sample of the same steel in the non-hardened state. In the case of embrittled steel, the etch will show up the grain boundaries of the parent austenite by blackening them, whereas there is no such reaction on the non-hardened steel. All the three reagents may not necessarily be suitable for every type of steel	
41. Picric acid-Zephiran chloride* solution	Picric acid Ethyl ether 1 N Zephiran chloride solution (12.8 per cent)	6 g 50 ml 2 ml	Dissolve picric acid in ether, add the water-Zephiran chloride mixture, stir well and then leave for 24 h to settle. For etching use only the clear part of the solution. Etching time several minutes with the specimen kept still. Next wash in an ether-alcohol solution, rewash with water and carefully wipe the dark deposit		
42. Picric acid in xylol-ethyl alcohol	Picric acid Xylol Ethyl alcohol	10 g 80 ml 20 ml	Add the alcohol immediately before use, filter the undissolved picric acid. Etching time : 30 minutes. Carefully wipe the dark deposit		

NOTE — The use of concentrated reagents is intended, unless otherwise specified.

*Zephiran chloride is a high molecular alkyl dimethyl benzyl ammonium chloride.

APPENDIX A

(Clause 9.3)

COMPARATIVE CHART OF GRIT NUMBERS OF
ABRASIVE PAPERS

A-1. For designation of grain sizes, of coated abrasives only grit numbers are recognized in IS : 715-1976*. Although grit numbers are now being universally adopted, coated abrasive papers are also available in a graded symbol sequence of 12/0 to 4½ covering the grit number range of approximately 600 to 12. There is also a special emery polishing paper for very fine polishing, such as used in preparation of metallurgical specimens, which is graded in a symbol sequence of 4/0 to 3, covering the grit number range of approximately 600 to 180. A comparative chart showing the relation between grit numbers and the customary grading symbols are given below for the guidance of users:

<i>Grit No.</i>	<i>Symbol for Silicon Carbide Paper</i>	<i>Symbol for Emery Polishing Paper</i>
600	12/0	4/0
—	—	3/0
500	11/0	2/0
460	10/0	0
360	—	—
320	9/0	½
280	8/0	—
240	7/0	1G
220	6/0	2
180	5/0	3
150	4/0	—
120	3/0	—
100	2/0	—
80	0	—
60	½	—
50	1	—
40	1½	—
36	2	—
30	2½	—
24	3	—

*Specification for coated abrasives (*third revision*)

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‘Pushpak’, Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001 550 13 46

‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road,
BANGALORE 560058 839 49 55

Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003 55 40 21

Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001 40 36 27

Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037 21 01 41

Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001 8-28 88 01

Savitri Complex, 116 G.T. Road, GHAZIABAD 201001 8-71 19 96

53/5 Ward No. 29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003 54 11 37

5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001 20 10 83

E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001 37 29 25

117/418 B, Sarvodaya Nagar, KANPUR 208005 21 68 76

Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road,
LUCKNOW 226001 23 89 23

NIT Building, Second Floor, Gokulpal Market, NAGPUR 440010 52 51 71

Patliputra Industrial Estate, PATNA 800013 26 23 05

Institution of Engineers (India) Building 1332 Shivaji Nagar, PUNE 411005 32 36 35

T.C. No. 14/1421, University P.O. Palayam, THIRUVANANTHAPURAM 695034 6 21 17

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